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PROTOCOL OF TEST METHODS FOR EVALUATING HIGH HEAT SINK FUEL THERMAL STABILITY ADDITIVES FOR AVIATION JET FUEL JP-8+100



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14. ABSTRACT

This report describes the high heat sink fuels thermal stability additive evaluation protocol of test methods as they apply to the evaluation of additives for JP-8+100. Individual test methods are described and a standardized methodology for test operation is presented. Acceptance criteria for both baseline fuels and candidate additives are also given.

15. SUBJECT TERMS

JP-8+100, additive, quartz crystal microbalance, isothermal corrosion oxidation tester, hot liquid process simulator, extended duration thermal stability test, advanced reduced scale fuel system simulator, carbon burnoff

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FOREWORD

This report describes the High Heat Sink Fuels thermal stability additive evaluation protocol of test methods as it applies to evaluation of JP-8+100 candidate fuel additives. It is intended to establish a method of standard evaluation for JP-8+100 additive candidates so that all additives receive an equal opportunity to be evaluated against standardized criteria. This protocol is intended as a basic guideline within which good sound engineering judgments can be made.

This report was compiled by Robert W. Morris, Jr. under project 3048, Task 304805, Work Unit 30480581 from input from each of the authors on their specific area of expertise.

Introduction:

The purpose of this document is to set forth the criteria that must be met in order for a candidate additive to be considered for use as a JP-8+100 thermal stability enhancing additive for aviation turbine fuels.

At the start of the JP-8+100 program in 1989, the Fuels Branch of the Air Force Aero Propulsion Laboratory solicited off-the-shelf additive candidates from all additive manufacturers who expressed an interest in the program. In all, over 300 additives were evaluated before the currently approved additive, BetzDearborn Spec-Aid 8Q462® was approved for use. Though the JP-8+100 development program is completed, there are some additives still under consideration as possible +100 additives. In addition, AFRL is currently seeking to take additized fuel technology to the next level through the development of additives for a generic High Heat Sink Fuel, JP-8+225.

The Approval Protocol – JP-8+100:

In order to be accepted as a +100-approved additive for the High Heat Sink Fuels program, candidate additives must exhibit superior performance in a series of tests. These tests range from small bench-scale tests to large scale simulator tests. Figure 1 shows the logical progression in which these tests are applied to the +100 additive approval process.

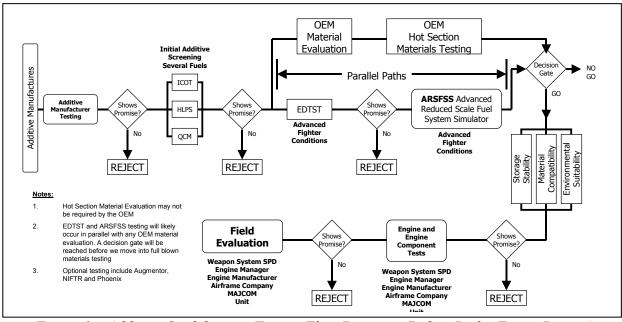


Figure 1 – Additive Qualification Testing Flow Diagram (Left to Right, Top to Bottom)

In order for a candidate additive to enter this process, the manufacturer/supplier must be reasonably confident, through their own in-house testing and evaluation, that the additive supplied has a reasonable expectation of successful completion of this process. If the additive performs satisfactorily in the small scale tests such as the Isothermal Corrosion Oxidation Tester (ICOT), Quartz Crystal Microbalance (QCM), and Hot Liquid Process Simulator (HLPS), then it

will be evaluated in larger scale devices like the Extended Duration Thermal Stability Test (EDTST). At the same time that the additive is evaluated in the EDTST, engine manufacturers (OEMs) are consulted with regard to any material evaluation requirements or issues. In some cases, OEMs may request or initiate hot section materials testing on the candidate additive. Since it may take a significant amount of time to complete OEM testing, any candidate additive having passed the EDTST and having no significant issues with material compatibility will proceed on to the Advanced Reduced Scale Fuel System Simulator (ARSFSS) for evaluation while the OEMs are considering what material compatibility testing may or may not be required.

Table 1 below summarizes the acceptance criteria for the additive protocol tests.

Table 1 – Summary of Acceptance Criteria for Baseline Fuels and Additive Candidates						
TEST METHOD	BASELINE FUEL SELECTION CRITERIA	GENERAL ACCEPTANCE CRITERIA				
QCM	Max deposition: 3 - 10 µg/cm² For information on currently accepted baseline fuels, contact AFRL	 ≤ 1.0 μg/cm² in an accepted baseline fuel OR At least as good as the Betz 8Q462 approved additive in the currently selected baseline fuel. 				
ICOT	 Deposition of between 50 and 200 mg/L. For information on currently accepted baseline fuels, contact AFRL 	Reduces deposition to < 20mg/L in any two out of three accepted baseline fuels				
HLPS	 Typical fuel available in the field (passes MIL-DTL-83133) Three types preferred: Hydrotreated Merox Treated Straight run 	 Delta-P <= 5 mmHg in 300 minutes Tube deposition (by carbon burn-off) not to exceed the Betz 8Q462 approved additive by direct comparison (slightly over if additive has no adverse effect on water separation) 				
EDTST	 At 350 °F bulk fuel and 500 °F wetted wall: Preheater max deposit >5 μg/cm² and <= 20 μg/cm² Heater tube max deposit >= 1000 μg/cm² 7-micron filter deposition <= 10,000 μg 	 At 375 °F bulk fuel and 500 °F wetted wall: Preheater max deposit <= 8 μg/cm² Heater tube max deposit <= 300 μg/cm² Cold Strip Visual – Clean to Slight Hot Strip Visual – Clean to Slight 				
ARSFSS ¹	Typical on-spec fuel	Meets (within 90%) or exceeds when compared to Betz 8Q462.				

Once an additive has successfully passed the ARSFSS it will be subjected to a full range of Material Compatibility, Storage Stability and Environmental Suitability evaluations. After successfully passing these evaluations, the additive is considered ready for full scale engine testing. Upon successful completion of engine testing, the additive can be used in a Field Evaluation Study, which will be implemented on actual flight aircraft at a selected base location(s) at the discretion of the Air Force.

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¹ At the time of this writing (April '02), detailed defined criteria not available for ARSFSS due to recent configuration changes. Typically, an additive candidate would receive a favorable review in the ARSFSS if it performed to within 85-90% of the currently approved additive in areas of tube deposition and valve hysteresis.

The remaining parts of this document will describe, in detail, the procedures and protocols used in each of the bench scale tests as well as the EDTST and the ARSFSS.

Quartz Crystal Microbalance (QCM)

Scope:

This method describes a technique in which a Quartz Crystal Microbalance System (QCM) is used to thermally stress aviation turbine fuel and determine the quantity of surface deposits produced. The purpose is to quantitatively evaluate the ability of an additive to prevent formation of surface deposits.

Summary of Test Method:

An aviation turbine fuel or aviation turbine fuel with additive is aerated and subsequently heated to 140°C for 15 hours. A quartz crystal microbalance is used to monitor the surface deposition produced. An oxygen sensor is used to monitor the oxidation process. Surface deposits produced on a quartz crystal oscillator lower its characteristic frequency. This frequency change is converted to a surface deposit in micrograms/cm² versus time.

Apparatus: See Figure 2.

Stainless steel reactor. The reactor consists of a 100 ml 316 stainless steel pressure vessel with access for the following: RF feed through, gas inlet tube, gas outlet tube, thermocouple, and pressure vent. The reactor is heated with a clamp-on band heater and the temperature is controlled by a PID controller with a thermocouple immersed in the liquid fuel. The controller must control the temperature to within $\pm 0.1^{\circ}$ C of the setpoint during the entire run. The heater must be capable of reaching and stabilizing at the test temperature in less than one hour. A magnetic stirrer and stir bar are used to minimize spatial temperature gradients.

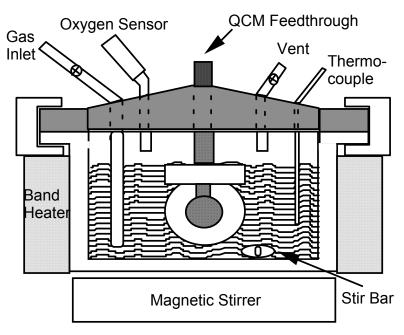


Figure 2 - Schematic drawing of the QCM apparatus.

Quartz crystal microbalance. The quartz crystals used are 5 MHz, 2.54 cm diameter, 0.33 mm thick, AT cut wafers. The crystals have gold electrode surfaces and are overtone polished. An

oscillator circuit tracks the impedance variations of the crystal, while a frequency counter measures the frequency to a precision of ± 0.1 Hz. A suitable clamp assembly is used to connect the crystal electrodes to the RF feed through of the reactor. The crystal is oriented vertically and completely immersed in the liquid fuel. Satisfactory crystals are available from Maxtek, Inc., Torrance, CA.

Oxidation monitoring. A polarographic oxygen sensor is used to monitor the oxygen level in the reactor headspace. This sensor should be approximately 0.5 inches in diameter to allow attachment to the reactor lid. The oxygen sensor should be capable of measuring oxygen levels in the reactor headspace from zero to 100 percent with a precision of ± 0.1 %.

Data acquisition system. A computer data acquisition system should be used which allows the following parameters to be recorded at one minute intervals: run time, crystal frequency, temperature, and headspace oxygen concentration.

Fuels:

In order to evaluate a thermal stability improving additive at least one suitable baseline fuel must be chosen. If possible, two baseline fuels should be available for these evaluations. Any baseline fuel used should have a final (15 hour) deposit between 3 and 10 μ g/cm². Contact AFRL for information on currently accepted baseline fuels.

Procedure:

The vessel is filled with 60±1 ml of aviation turbine fuel or aviation turbine fuel with additive, and the reactor lid closed. The fuel is sparged for one hour with air to insure that the fuel is air saturated. After sparging is complete the sparge gas is turned off and the gas inlet/outlet valves are closed at ambient pressure. At this point the heater, which is set at 140°C, is turned on and computer data acquisition is begun. The run time, crystal frequency, reactor temperature, and headspace oxygen concentration are monitored and recorded at one minute intervals by the data acquisition system. Runs are conducted for 15 hours; at the end of this time the heater is turned off. After the reactor has cooled to room temperature, it is opened and the fuel and crystal are discarded. All surfaces that contact the fuel are cleaned repeatedly with an equivolume mixture of toluene, acetone, and methanol with laboratory wipes, cotton-tipped swabs, and pipe cleaner brushes, until brown deposit residue is completely removed. The reactor must be completely dry before another fuels is tested. The crystal is replaced with a new, unused crystal prior to each run.

Data analysis:

The equation used to convert frequency change to mass accumulation is:

$$\rho_s = -\left(2.21 \times 10^5 \text{ g/(cm}^2\text{s})\right) \frac{\Delta f}{f_0^2}$$

where f_0 is the initial resonant frequency, Δf is the change in frequency (i.e., the current frequency – the initial frequency), and ρ_s is the area surface mass density (mass/area). The initial frequency, f_0 , is chosen by observing the temperature-time heat-up history of the run (See Fig. 3). The initial time, t_0 , is when the reactor stabilizes at the run temperature (i.e., within 0.2°C of the temperature controller set point). Frequency points that are not within 0.2°C of the setpoint are

discarded. The initial frequency is the measured frequency at the initial time. The above equation is used to convert the measured frequencies into areal mass density versus time.

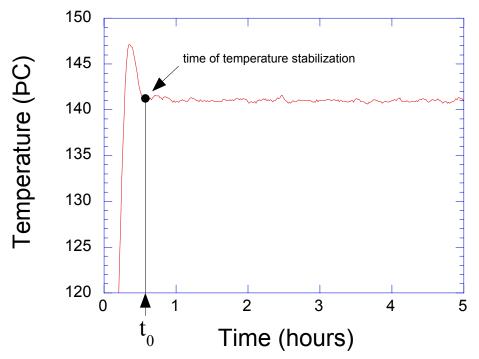


Figure 3 - Plot Showing Determination of Initial time, t_0 , for Determination of Initial Frequency, f_0 .

For example, the frequency at 15 hours is used to calculate the final deposition as follows: for an initial frequency of 4.988300 MHz and a final frequency of 4.988100 MHz,

$$\rho_s = -\left(2.21 \times 10^5 \text{ g/(cm}^2\text{s})\right) \frac{4,988,100 - 4,988,300\text{s}^{-1}}{4,988,300^2 \text{s}^{-2}} = 1.78 \mu\text{g/cm}^2.$$

Data reporting:

The deposition level at 15 hours is to be reported. Graphs of deposition and headspace oxygen level versus time may be required.

Proposed criteria for approval:

The additive must reduce the final deposition to $\leq 1.0 \,\mu\text{g/cm}^2$ in any suitable baseline fuel.

As a possible evaluation of antioxidants, report the time at which half the oxygen is consumed. This will require a fast oxidizing fuel which completely oxidizes in < 3 hours.

Isothermal Corrosion Oxidation Tester (ICOT)

Scope:

The Isothermal Corrosion Oxidation Tester (ICOT) is used to evaluate the effectiveness of thermal stability additives in aviation turbine fuel by stressing the baseline fuel and the additized fuel at a constant temperature in the presence of flowing air. The fuel is filtered after it has cooled. The thermal stability of the fuel is measured by the amount of solids collected on a filter.

Summary:

Operating conditions require 100 ml of the fuel sample to be stressed at 180° C with air flowing at 1.3 L/hr for a duration of 5 hours. The fuel sample is completely cooled then filtered through a 0.7 to 1 μ m glass microfiber filter. Solids are measured gravimetrically. The thermal stability of the baseline fuel and the baseline fuel with the thermal stability additive is based on the amount of solids collected on the filter. The thermal stability additive is considered acceptable based on the total solids produced.

Apparatus:

See ASTM Method D4871-88 for a description of the apparatus. The filter shall be a glass microfiber filter with a particle retention of 0.7 to $1 \mu m$.

Materials:

Not all fuels are suitable for evaluating the thermal stability additive. Some fuels, such as JPTS, do not produce significant deposition (< 20 mg/L) at these testing conditions. On the other hand, some fuels will produce copious amounts of deposits (> 200 mg/L). These fuels are generally fast oxidizing fuels that may benefit for a short period of time from the thermal stability additive. However, over a five hour period either the additive package will be consumed or the amount of insolubles generated will be so great that the ability of the additive to influence thermal stability will be exceeded. So, the required fuel for this test is a fuel that produces a moderate amount of deposits (between 50 and 200 mg/L). Three suitable fuels must be used to evaluate a thermal stability additive. Contact AFRL for information on currently accepted baseline fuels.

Preparation:

A new or clean glass inlet tube must be used for each experiment. If the gas inlet tubes are to be reused they must be cleaned in a glass annealing oven. The following oven temperature program should be used: begin with an initial temperature of 200°C then ramp the temperature to 600°C at a rate of 7°C/minute then cool. The test cells should be cleaned in the following sequence: wash with soap and water, rinse with acetone, dry residue liquid, soak with a strong alkaline or acidic glass cleaning solution for two hours, rinse with distilled water then allow to dry.

Test Procedures:

a. Turn on the heating block and allow it to stabilize at a temperature of 180°C (approximately 45 minutes). Turn on water recirculation bath for condensers at a temperature of 15°C.

- b. Measure 100 ml each of the baseline fuel and the fuel with the thermal stability additive. Pour each fuel into a test cell.
- c. Place test cell into test well. Attach condenser. Insert glass inlet tube.
- d. Attach gas supply tubing and adjust the air flow to 1.3 L/hr.
- e. Allow test to run for 5 hours.
- f. After 5 hours remove test cells from wells. Remove the gas inlet tubing. Leave the condenser rods in the test cells. Allow test cells to sit at room temperature for a minimum of 16 hours to a maximum of 24 hours.
- g. Pre-weigh a filter for each sample, and one control filter. The control filter is subjected to the same rinsing process as the others, and the change in its weight before and after rinsing is used to distinguish between real weight changes caused by ICOT deposits and weight changes caused by the rinsing process. Weight changes caused by rinsing are usually relatively small. Filter each fuel by vacuum filtration. Rinse test cells with a reagent grade typical straight chain hydrocarbon solvent with a boiling range between pentane (37°C) and octane (126°C), such as hexane or heptane. Rinse filter with the same hydrocarbon solvent. Note: Sometimes the deposits will adhere to the surface of the test cell. In this case place some hydrocarbon solvent into the test cell and put test cell in an ultrasonic bath for several minutes as needed until deposits are loosened.
- h. Place filter in a petri dish, tilting the lid. Heat filters in a oven at 100°C for a minimum of 3 hours. Place the petri dish in a desiccator until it is completely cool.
- i. Re-weigh the filter.

Calculations:

Report the amount of deposits in mg of solid per liter of fuel using the following formula:

((weight of filter with deposits in mg) - (weight of filter in mg) + (weight of control filter before rinsing in mg) - (weight of control filter in mg after rinsing in mg)) / 0.1 liter

Proposed criteria for approval:

The thermal stability additive is considered acceptable if it reduces deposition to below 20 mg/L in two out of the three baseline fuels.

Hot Liquid Process Simulator (HLPS):

Scope:

The Hot Liquid Process Simulator (HLPS) is an accelerated, bench-scale, dynamic flowing-type test that subjects the fuel to conditions that can be related to those in gas turbine engine fuel systems. A special test procedure developed by P&W has proven useful as a screening test for evaluating the effectiveness of candidate thermal stability improving additives in aviation turbine fuels. When used in conjunction with carbon burn-off, this method can be effectively used to quantitatively rank additive performance based on micrograms per square centimeter ($\mu g/cm2$) of carbon formed on a stainless-steel tube.

Summary of Test Method:

The HLPS is similar in operation to the Jet Fuel Thermal Oxidation Tester (JFTOT) employed in ASTM D 3241-01. The HLPS differs from the JFTOT in its modular design, extended temperature range, flow rate capabilities and accommodation of greater fuel volumes. The test conditions selected to evaluate additive performance are much more severe than those specified in the standard JFTOT procedure. Tests are performed at 335°C (635°F) for 5 hours. Series 316 stainless-steel tubes are substituted for conventional aluminum tubes to permit quantification of the deposit by carbon burn-off. Carbon burn-off is accomplished using a LECO RC-412 Carbon Analyzer.

The fuel is pumped at a fixed volumetric rate of 3.0 milliliters per minute (mL/min.) over a resistively heated Series 316 stainless-steel tube. Subsequently, the fuel flows though a 17-micron (μ) nominal-porosity precision stainless-steel filter where fuel degradation products may become trapped. Additive effectiveness is derived from the amount of deposit formed on the 316 stainless-steel heater tube and the rate of plugging of the 17-μ stainless-steel filter.

Apparatus:

A photo of the HLPS is shown Figure 4. A schematic of the internal workings of the test device is shown in Figure 5. See ASTM D3241-01 for a detailed description of the JFTOT. The HLPS and the JFTOT are manufactured by Petroleum Analyzer Company (PAC) of San Antonio, TX.

Test Fuels:

Several fuels of varying quality and processing history should be used to demonstrate the ability of a candidate additive to improve thermal stability. A hydrotreated fuel, a merox-treated fuel, and a straight-run fuel provide a reasonable cross section of fuel types. Initial screening tests should be performed using a well-documented reference fuel. The reference fuel should be typical of a fuel found in the field that just meets the 260°C (500°F) thermal stability requirement defined in Mil-DTL-83133 for JP-8 fuel.

Test Procedure:

1. Follow the procedure as described in ASTM D3241-01 with the exception of the test conditions listed below, which have been modified for use in evaluating additive performance.

a. Test Temperature: 335°C (635°F)

b. Test Duration: 300 minutes

c. Heater Tube: 316 Stainless Steel

d. Fuel Quantity: 1000 mL

2. Carefully monitor differential pressure (ΔP) across the 17-μ stainless-steel filter. If the additive produces bulk fuel insolubles that begin to plug the 17-μ filter, bypass at 300-millimeters (mm) Hg and complete the 300-minute test.

Tube Preparation Prior To Burning Off (Quantification) of Deposit:

- 1. At the conclusion of the test, carefully remove the stainless-steel heater tube from the heater-tube housing. Store the tube wet with fuel in the plastic tube it originally came in. The tube is stored wet with residual fuel to preserve the deposit and prevent drying out and possible loss of the heavier deposits. If carbon analysis is to be performed immediately, this precaution is not necessary.
- 2 Prior to analysis, the larger diameter "shoulders" of the heater tubes are cut off using an oil-cooled diamond cut-off wheel. A LECO VC-50 precision diamond saw, model 801-900 or equivalent has been shown to be satisfactory for this purpose. The cut is made on the larger-diameter shoulder approximately 2 mm from the end of the small diameter test-portion of the tube. After both shoulders have been removed, the tube is handled using clean, small-pointed tweezers. Tweezers are used to prevent contamination of the heater tube with carbon from fingerprints. Grasp the tube with one small tweezer-point inserted into the thermocouple hole and the other on the remaining 2-mm shoulder section. The tube is then rinsed with hexane or heptane to remove residual fuel and cutting-wheel debris. Carefully rinse the thermocouple hole as well as the entire length of the tube. Dry the tube with a gentle stream of compressed-nitrogen gas. After cleaning, the prepared heater tube is handled only with tweezers and caution should be taken in ensuring the tube does not contact any other surface prior to analysis.

Tube Rating Procedure

Fuel deposits on the prepared 316 series stainless-steel tube are quantified using carbon burn-off as described in Appendix A.

Reporting:

Report the following for each baseline and additized fuel:

a. Total quantity of carbon deposit on the heater tube as measured by carbon burn-off measured in $\mu g/cm^2$

$$\frac{total\mu g}{6.02012cm^2} = \frac{\mu g}{cm^2}$$

b. Differential pressure in mm Hg across the 17-μ filter

Criteria for Determining if the Additive Merits Elevation to Second Level Testing

- 1. $\Delta P < 5$ mm Hg in 300 minutes
- 2. Deposition not to exceed current state-of-the art as determined by a direct comparison with Betz 8Q462 using the same baseline reference fuel. If the additive being evaluated is known to have no adverse effects on filter separators, then special consideration should be given to allow a modest increase in deposit over that of Betz 8Q462.

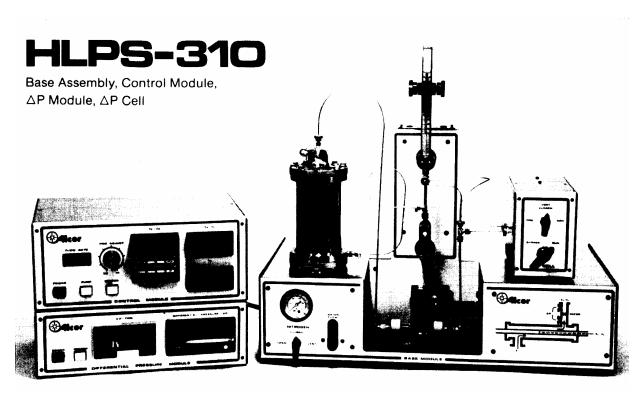


Figure 4 - Hot Liquid Process Simulator

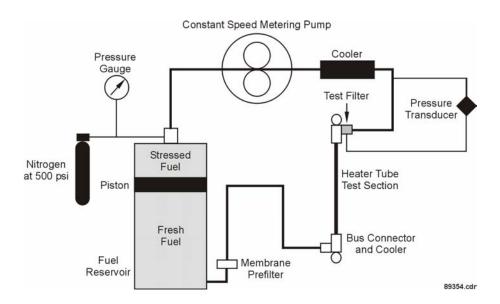


Figure 5 - Schematic of Hot Liquid Process Simulator

Extended Duration Thermal Stability Test (EDTST)

Scope:

This section describes the Extended Duration Thermal Stability Test (EDTST) and its application in the evaluation of High Heat Sink Fuel additive candidates. The purpose of the EDTST is to simulate the fuel flow and heat cycle characteristics of a typical aircraft fuel system and to evaluate fuels and additives under these conditions. Since this test does not operate at conditions that accelerate thermal degradation (high temperature, long residence times), longer duration times are necessary to evaluate fuels and additives.

Summary:

The EDTST subjects the fuels to specified bulk fuel and wetted wall temperatures at residence times related to those occurring in gas turbine fuel systems. A fuel bypass line is incorporated to represent Military aircraft designs for thermal management that recirculate fuel from the engine back to the airframe tanks. Also, the fuel is exposed to the specified wetted wall temperatures for very short durations and then is scraped. This is representative of the fuel exposed to the engine injection nozzles.

Apparatus:

The EDTST system consists of a 60 gallon feed tank, an electrical motor driven gear pump, two clamshell furnace heaters, and a scrap tank. A schematic of the EDTST system is shown in Figure 6. The first furnace heater (preheater) in the system is used to establish the desired fuel bulk temperature into the second heater and to establish the desired fuel bypass temperature. The fuel bulk temperature represents the temperature that results from aircraft and engine heat loads. JP8+100 fuel will provide a capability to operate at or above 375°F (204°C) bulk fuel temperatures.

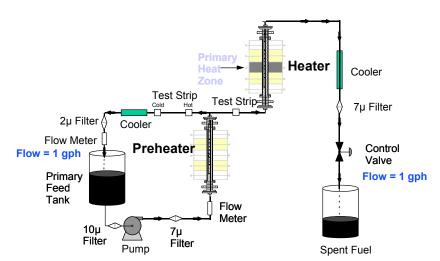


Figure 6 - EDTST Rig Schematic Diagram

Another requirement for JP8+100 fuel is to provide a wetted wall temperature capability of 500°F (260°C) for engine fuel injection nozzle design. The temperature is established in the second furnace heater (main heater) to represent the wetted wall temperatures associated with engine injection nozzles.

Both furnace heaters are 0.81 meters long and resistance heated. They each have 5 heating element zones that are independently controlled. The fuel flows upward through a single stainless steel tube in each heater. The tube in the preheater has an O.D. of 1.27 cm and a wall thickness of 0.0889 cm. The tube in the main heater has an O.D. of 0.32 cm and a wall thickness of 0.0889 cm. Each tube is assembled inside a thick walled furnace tube that has an I.D. of 2.54 cm and an O.D. of 5.08 cm. The tubes have thermocouples attached to the outer wall for measuring wetted wall temperatures. The annular space between the furnace tube and heater tubes is filled with sand. A typical main heater assembly is shown in Figure 7. A fuel bypass line is installed downstream of the preheater to represent the aircraft recirculation line from the engine to the airframe tanks. A water/fuel cooler is installed in this line to represent the aircraft ram air heat exchanger. A 2µ filter is also installed in the line for 4 hours to measure particles in the recirculated bulk fuel. Since effects of recirculation are one of the purposes of this test, the filter is installed only for a short duration. Aircraft fuel systems will probably not have a filter in the recirculation line. A 7µ filter is also installed downstream of the heater. This filter provides an indication of particles that the fuel nozzles will experience in the advanced system design with a heat exchanger downstream of the engine fuel controls. Witness strips in housings are located after the preheater and immediately in front of the bypass line heat exchanger.

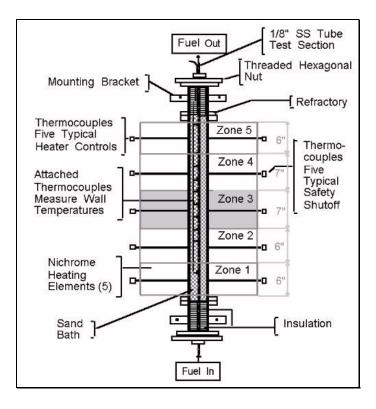


Figure 7 - EDTST Main Heater Assembly

Procedure:

The feed tank is filled with 55 gallons of fuel to be tested. The pump is turned on to establish flow of 1 gallon per hour in both the preheater bypass line and though the heater. The system heaters are then turned on to establish a bulk temperature of 375°F out of the preheater and a maximum wetted wall temperature of 500 °F on the heater tube. When these conditions are reached, the test is conducted for 96 hours. The feed tank is refilled at the 48-hour test interval. After 96 hours the heaters are turned off and the heater clamshells are opened to cool down the system. After the system is cool, the pumps are turned off and the system is partially disassembled. The preheater, heater and heat exchanger tubes are cut up in segments for LECO analysis as described in Appendix A. The 2 and 7-micron filters are also subjected to LECO analysis. The witness strips shall be inspected and appearance due to deposition shall be noted systems. The EDTST system is computer controlled and can run unattended for long periods of time.

Baseline Fuel Selection

To select fuels as baseline, tests shall be conducted at 350°F bulk fuel and 500°F wetted wall temperature conditions. A fuel will be considered acceptable for use as a baseline fuel if, after a 96-hour test run without additive, it meets the following criteria:

- Preheater tube segment maximum deposition of > 5 and ≤ 20 , $\mu g/cm^2$
- Heater tube segment maximum deposition of $\geq 1000 \, \mu \text{g/cm}^2$
- Deposition in the 7-micron filter of $\leq 10,000 \mu g$

Reporting:

Report the following for each baseline fuel and additized fuel. Table 2 shows an example of how typical EDTST can be reported.

- a) The quantity of carbon deposition on the preheater, heater, and heat exchanger tube segments. The deposit per tube segment is divided by the surface area and reported in units of $\mu g/cm^2$. Typical plots used to report the results are shown in Figures E3, E4 and E5. It should be noted that the bulk temperature was 350°F for the JP-8 Fuel and 400°F for the JP-8+100 (Betz 8Q462, POSF-3549) fuel for the plots.
- b) The amount of the filter deposits in units of μg 's.
- c) Inspection of the witness strips shall be conducted and their appearance in regards to deposition shall be noted.

Table 2 – Typical EDTST Data Report

Additive ID	Max Segment Deposit, μg/cm ²			Filter (μg)		Strips (Visual)	
(Fuel ID)	Heater	Preheater	Heat Exch.	2μ	7μ	Cold	Hot
Ex. EC212483C (POSF-3860)	227	32*	116	184	43,050	Slight	Heavy*
Ex. EC212483D (POSF-3861)	84	44*	127	310	20,450	Slight	Heavy*
Acceptable Limits	300	8	Report	Report	Report	Clean to Slight	Clean to Slight

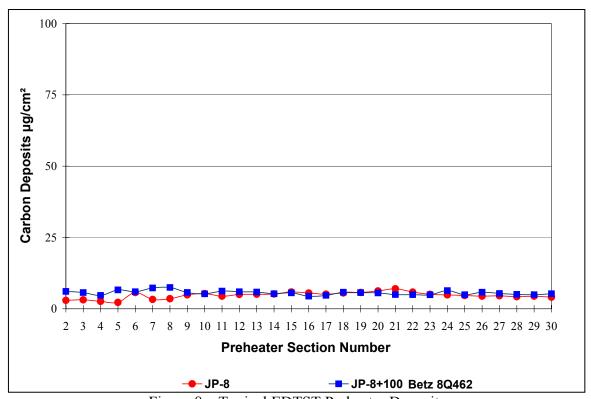


Figure 8 – Typical EDTST Preheater Deposits

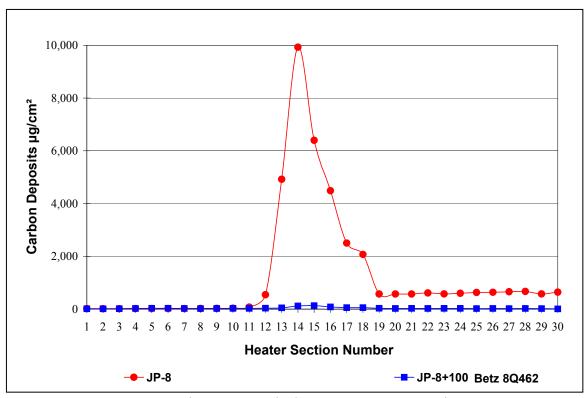


Figure 9 – Typical EDTST Heater Deposits

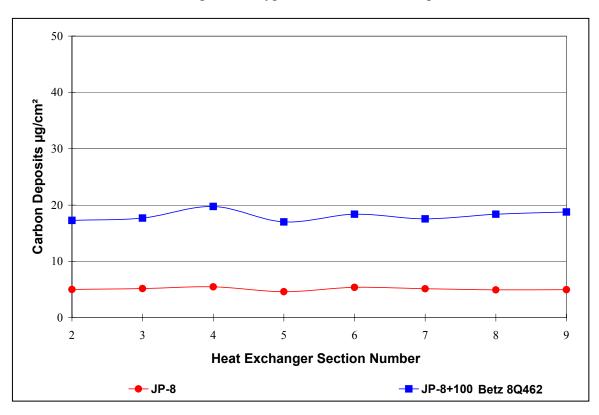


Figure 10 – Typical EDTST Heat Exchanger Deposits

Criteria for Approval of Additive Candidate for Elevation to ARSFSS Evaluation (Table 3): Table 3 – EDTST Approval Criteria

Additive ID	Max Segment Deposit, μg/cm ²		Filter (µg)		Strips (Visual)		
(Fuel ID)	Heater	Preheater	Heat Exch.	2μ	7μ	Cold	Hot
Acceptable Limits	300	8	Report	Report	Report	Clean to Slight	Clean to Slight

Advanced Reduced Scale Fuel System Simulator (ARSFSS)

Scope:

The Advanced Reduced Scale Fuel System Simulator (ARSFSS) is designed to closely simulate the hardware, thermal and fuel flow characteristics of an aircraft fuel system. It provides the last analysis of potential High Heat Sink Fuel additives prior to going into actual engine testing.

Summary:

The Advanced Reduced Scale Fuel System Simulator was designed to realistically simulate the thermal and flow profiles of advanced aircraft. The simulator consists of three integrated subsystems: 1) the fuel conditioning system, 2) the airframe fuel system, and 3) the engine fuel system. A schematic of the simulator is shown in Figure 11. The simulator is currently configured to simulate the F-22 aircraft with the F119 engine. The fuel flow established in the simulator is 1/72 scale of the F119 engine and the burn flow is 1/3 of the flow for a single F119 fuel nozzle. The total fuel required for each test is approximately 1,500 gallons.

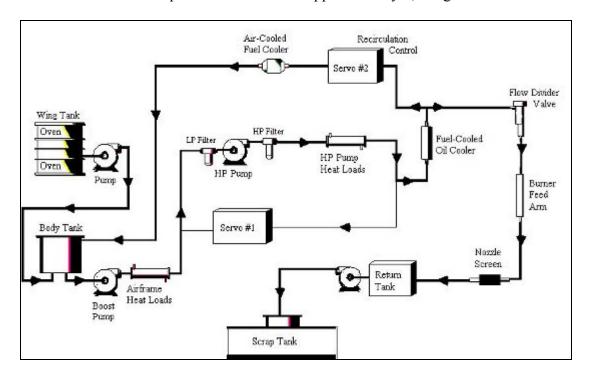
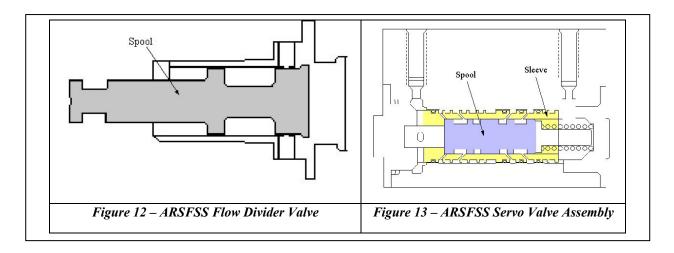


Figure 11 – Schematic of Advanced Reduced Scale Fuel System Simulator (ARSFSS)

Apparatus:

Actual engine hardware has been incorporated into the engine portion of the simulator to evaluate the impact of fuel deposits on component performance. The specific test articles include a flow divider valve and two servo valves. The flow divider valve (Figure 12) is an actual F119 valve that has been modified by changing the slot width for the reduced flow. The servo valves

(Figure 13) are modified F119 engine control valves. They are the second stage valves of electrohydraulic valves used in the engine control system. The materials, clearances and function is representative of the valves on the F119 engine. The performance of these valves is determined by hysteresis and visual inspection. Simulations of the fuel-cooled oil cooler (FCOC) and the burner feed arm (BFA) are also incorporated to study thermal stability effects. The FCOC represents the engine lube system cooler. It consists of an induction heater and a steel manifold with three 3/8" tubes and associated thermocouples. The tubes are connected and provide for three passes through the heater. The tube that is used for the final pass is removed after each test. It is cut into 2 inch segments and subjected to carbon analysis.



The burner feed arm is RF induction heated. It consists of a steel clamshell with a 1/8 inch stainless steel tube installed in middle of the clamshell. Thermocouples on the outside of the tube are positioned along the entire length to measure the temperature profile of the tube. At the end of the tests, this tube is cut up into 1 inch segments and subjected to carbon analysis as well.

A generic F119 mission profile was developed based on actual flight conditions and used to evaluate the deposit tendencies of jet fuel. A comparison of the deposits produced in the BFA with JP-8 and JP-8 + Betz 8Q462 is shown in Figure 14. A bulk fuel temperature of 350°F (177°C) out of the FCOC and a wetted wall temperature of 500°F (260°C) in the BFA were the selected test conditions. As can be seen in Figure 14, the carbon deposits have been dramatically reduced with the additized fuel. Significant hysteresis was measured in both the flow divider valve and the servo valve with JP-8 while none was measured with JP-8 + Betz 8Q462. Visual inspection of the valves revealed a thin lacquer layer had formed on the critical internal surfaces which were believed responsible for the hysteresis.

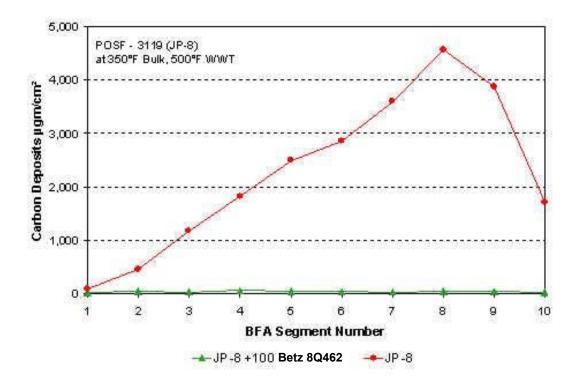


Figure 14 – Carbon Burnoff of ARSFSS Burner Feed Arm Segments for JP-8 and JP-8+100

Data Reporting:

The following data will typically be reported:

- FCOC carbon burnoff
- BFA carbon burnoff
- Visual appearance of valves and components
- Hysteresis measurements on servo valves and flow divider valves
- Any other pertinent data

Appendix A

Carbon Burnoff Method for Measuring Carbon Deposition On Materials

Scope:

This method describes a technique for measuring carbon deposits on metal surfaces by carbon burnoff

Summary:

Carbon burnoff is a technique to measure carbon deposits on metal surfaces produced during the exposure of samples to heated aviation turbine fuel. The deposits are measured by heating the samples to 650 °C in a stream of oxygen and measuring the amount of CO_2 produced. 650 °C is sufficient to oxidize surface carbon, but not high enough to remove carbon from stainless steel or similar alloys. This technique is incompatible with carbon-containing substrates that have oxidizable carbon at \leq 650 °C. Typical samples include metal tubing with deposits on the inside of tubes (EDTST, NIFTR), metal cylinders with deposits on the outside of the cylinder (JFTOT), or metal-mesh filters with deposits dispersed throughout the metal mesh (EDTST).

Apparatus:

A Leco RC-412 Multiphase Carbon Determinator (manufactured by Leco Corporation, St. Joseph, MI) is an approved apparatus for this method. Other devices may be equivalent. This procedure describes the use of the Leco Multiphase Carbon Determinator, but may be adapted for use with other devices with AFRL/PRTG permission. At a minimum, such an alternate device should include a controllable furnace/flow cell, a catalyst to convert any CO to CO₂, and sample and reference IR cells sensitive specifically to CO₂. The minimum detection level shall be 10 µg of carbon or less.

Sample Preparation:

Samples should be prepared in such a way as to minimize, as much as possible, surface contaminants such as fingerprints and the presence of fuel in the materials to be tested, since contaminants may contain carbon and will bias the measurement. Sample preparation procedures shall be outlined in various methods (such as EDTST or JFTOT), but at a minimum should involve thorough rinsing of the materials in a clean, volatile hydrocarbon solvent, such as heptane, and subsequent drying of the material in a vacuum oven. Drying should be sufficient to assure complete removal of solvent and fuel. Sample size for the Leco carbon analyzer are constrained to ≤ 2 inches in length and 0.5 inches in thickness.

Test Procedure:

The dried samples are placed in the sample holder (e.g., a clean and dry quartz "boat") and inserted into the Leco's furnace zone. The sample is then heated to 650 °C in a stream of pure oxygen. The amount of CO_2 evolved is measured as a function of time with an IR cell, with the measurement terminating when the CO_2 level returns to baseline. The weight of CO_2 evolved is found by integrating the CO_2 vs. time signal.

The weight of carbon evolved from the sample is found by multiplying the weight of CO_2 by the fraction of C in CO_2 . The fraction of carbon (MW=12.01) in CO_2 (MW=44.01) is 0.273.

$$\mu g C = (\mu g CO_2) X (0.273)$$

Samples containing large amounts of carbon (mg levels) or large surface areas shall be ramped at 40 °C/minute to 650 °C, to avoid uneven or incomplete carbon burnoff. Explosions and visible flames shall invalidate the test sample, since they can lead to inaccurate measurements. Care must be taken to ensure that all of the evolved gas flows through the CO₂ detector. Blank samples that are specific to the test method (see test methods for blank procedure) shall be measured and reported. Calibrations shall be performed in accordance with the apparatus standards. Calibration data shall be reported.

Results:

Results shall be reported as described in the test method (e.g., JFTOT or EDTST), typically in μg of carbon per sample.